ICS 73.080
CCS D 52

NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

GB/T 3286.2-2025

Replacing GB/T 3286.2-2012

Methods for chemical analysis of limestone and dolomite - Part 2: Determination of silicon and aluminium content

石灰石及白云石化学分析方法 第2部分：硅、铝含量的测定

Issued on: August 29, 2025 **Implemented on: March 1, 2026**

Issued by: State Administration for Market Regulation;
Standardization Administration of the PRC

Table of Contents

1 Scope

This document describes the determination of silicon dioxide content using the silicomolybdenum blue spectrophotometric method, perchloric acid dehydration gravimetric method, inductively coupled plasma atomic emission spectrometry (ICP- AES); as well as the determination of alumina content using the chromium azurite S spectrophotometric method, complexometric titration, ICP-AES.

This document is applicable to the determination of silicon dioxide and alumina content in limestone, dolomite, metallurgical lime. The silica-molybdenum blue spectrophotometry has a determination range (mass fraction). for silica content 0.050% ~ 4.00%; the perchloric acid dehydration gravimetric method has a determination range (mass fraction). silica content > 2.00%; the inductively coupled plasma atomic emission spectrometry has a determination range (mass fraction). silica content 0.070% ~ 3.50%;

the chromium azure S spectrophotometry has a determination range (mass fraction).

alumina content 0.010% ~ 0.75%; the complexometric titration has a determination range (mass fraction). alumina content > 0.50%; the inductively coupled plasma atomic emission spectrometry has a determination range (mass fraction). alumina content 0.040% ~ 1.00%.

2 Normative references

GB/T 2007.2

GB/T 6379.1

GB/T 6379.2

GB/T 6682

GB/T 8170

GB/T 12806

GB/T 12807

GB/T 12808

JJG 768

3 Terms and definitions

This document does not contain any terms or definitions that require definition.

4 Determination of silicon content. Silicomolybdate blue

spectrophotometric method

4.1 Principle

The sample is melted with anhydrous sodium carbonate-boric acid mixed flux, leached with dilute hydrochloric acid. A portion of the solution is taken; in approximately 0.15 mol/L hydrochloric acid medium, ammonium molybdate reacts with silicic acid to form a silicomolybdate heteropoly acid. An oxalic acid-sulfuric acid mixture is added, to eliminate interference from phosphorus and arsenic. The solution is then reduced to silicomolybdate blue with ferrous ammonium sulfate solution. The absorbance is measured at 680 nm using a spectrophotometer.

4.2 Reagents

Unless otherwise specified, only approved analytical grade reagents and distilled water of grade II or higher purity conforming to GB/T 6682 are used in the analysis.

4.2.1 Mixed flux. Grind two parts of anhydrous sodium carbonate with one part of boric acid (mass ratio 2.1); mix thoroughly.

4.2.2 Hydrochloric acid, rho approx. 1.19 g/mL.

4.2.3 Sulfuric acid, rho approx. 1.84 g/mL.

4.2.4 Anhydrous ethanol, rho approx. 0.80 g/mL.

4.2.5 Hydrochloric acid, 1 + 5.

4.2.6 Hydrochloric acid, 1 + 14. 4.2.7 Sulfuric acid, 1 + 8.

4.2.8 Ammonium molybdate solution, 60 g/L, stored in a plastic bottle, filtered before

use if necessary.

4.2.9 Oxalic acid-sulfuric acid mixture. Weigh 35 g of oxalic acid (H2C2O4-2H2O);

dissolve in 1000 mL of sulfuric acid (4.2.7).

4.2.10 Ferrous ammonium sulfate solution, 60 g/L. Weigh 6 g of ferrous ammonium

sulfate [Fe(NH4)2(SO4)2-6H2O]; dissolve in water containing 3 ~ 5 drops of sulfuric acid (4.2.3); then dilute with water to 100 mL. Prepare it before use.

4.2.11 Silica standard solution.

4.2.11.1 Weigh 0 2500 g of silica (content not less than 99.99%, ignited at 950 °C ~ 1000 °C for 30 min before weighing and cooled to room temperature) into a platinum crucible. Add 3 g of mixed flux (4.2.1); mix well; then cover with 1 g of mixed flux (4.2.1). Cover with a platinum cap (leaving a gap); place the platinum crucible in a 950 °C high-temperature furnace for melting for 10 min. Remove and cool to room temperature. Place the platinum crucible and platinum cap in a polytetrafluoroethylene beaker containing 100 mL of hot water; heat at a low temperature to leach the molten material until the solution is clear. Wash the platinum crucible and platinum cap with hot water and cool to room temperature. Transfer the solution to a 500 mL volumetric flask; use water to dilute it to the mark; mix well; immediately transfer to a plastic bottle for storage. 1.00 mL of this solution contains 500.0 μg of silica.

4.2.11.2 Transfer 50.00 mL of silica standard solution (4.2.11.1) to a 500 mL volumetric flask; use water to dilute it to the mark; mix well; immediately transfer to a plastic bottle.

1.00 mL of this solution contains 50.0 μg of silica.

4.2.11.3 Transfer 20.00 mL of silica standard solution (4.2.11.1) to a 500 mL volumetric flask; use water to dilute it to the mark; mix well; immediately transfer to a plastic bottle.

1.00 mL of this solution contains 20.0 μg of silica.

4.3 Instruments and equipment

The analysis shall use common laboratory instruments and equipment. One-mark pipettes,

graduated pipettes, one-mark volumetric flasks shall comply with the provisions of GB/T 12806, GB/T 12807, GB/T 12808.

4.3.1 Analytical balance, graduation value 0.1 mg. 4.3.2 Spectrophotometer.

4.3.3 Platinum crucible, 30 mL.

4.4.1 Prepare specimens according to GB/T 2007.2.Specimens shall be processed to a

particle size of less than 0.125 mm.

4.4.2 Limestone and dolomite specimens shall be dried at 105 °C ~ 110 °C for 2 h before analysis, then cooled to room temperature in a desiccator.

4.4.3 The preparation of metallurgical lime specimens shall be carried out quickly. After

preparation, the specimens shall be immediately sealed in ground glass bottles or plastic bags and stored in a desiccator. Specimens shall not be dried before analysis and shall be analyzed as soon as possible.

4.5.1 Number of determinations

For the same specimen (4.4.2 or 4.4.3), at least two independent determinations shall be performed.

4.5.2 Sample amount

Weigh 0.50 g of specimen, accurate to 0 0001 g. For metallurgical lime specimen, the specimen shall be weighed quickly.

4.5.3 Blank test

Perform a blank test along with the sample.

4.5.4 Sample decomposition

4.5.4.1 Place the sample (4.5.2) in a platinum crucible pre-filled with 3.0 g of mixed flux (4.2.1); mix well; then cover with 1.0 g of mixed flux (4.2.1). Place the platinum crucible in a high-temperature furnace at a temperature below 300 °C; cover with a platinum lid (leaving a gap). Gradually raise the furnace temperature to 950 °C ~ 1000 °C and melt for 10 minutes. Remove; rotate the platinum crucible; cool it.

4.5.4.2 Rinse the outer wall of the platinum crucible with water. Place the platinum crucible and platinum lid in a 300 mL beaker; add 75 mL of hydrochloric acid (4.2.5);